

powder having a thermal diffusivity of from about 0.15 cm<sup>2</sup>/s to about 0.20 cm<sup>2</sup>/s, as claimed in the above-identified application (Clere Declaration ¶ 4).

*Shaffer I and II*

In particular, Shaffer I and II, which have the same disclosure, relate to a method of forming pellets or agglomerates of high density boron nitride (BN) by crushing high purity hexagonal boron nitride (hBN) into BN particles extending over a size range of at least 100 µm with a majority of the particles having a particle size above 50 µm and cold pressing the crushed particles into a compacted form (see Abstract) (Clere Declaration ¶ 5). The compacted form is then granulated and again cold pressed to form pellets or agglomerates of BN particles, which can then be crushed into a powder (see Abstract) (Clere Declaration ¶ 5).

In particular, in Example 1, Shaffer I and II teach that high purity BN was crushed to give a crushed powder having an oxygen content of 0.426 %, a surface area of 2.51 m<sup>2</sup>/g, a tap density of 0.92 g/cc, and 0.09 % soluble borates (see Shaffer I at col. 4, lines 45-48 and Table II) (Clere Declaration ¶ 6). The crushed powder had a particle size ranging from about 44 µm (-325 mesh) to greater than 420 µm (+40 mesh) (Shaffer I, Table II) (Clere Declaration ¶ 6). These references then teach that the crushed powder was compacted using a horizontal press at a pressure of 19,000 psi (Shaffer I, col. 4, lines 48-49) (Clere Declaration ¶ 6). The compacted pieces were then granulated by forcing the material through a screen with openings of approximately ½ inch (Shaffer I, col. 4, lines 49-51) (Clere Declaration ¶ 6). The granulated particles were again compacted at 19,000 psi giving a resultant product with a density of 1.91 g/cc (Shaffer I, col. 4, lines 51-54) (Clere Declaration ¶ 6). Thus, Shaffer I and II teach: (1) crushing high purity BN; (2) compaction of the crushed BN; (3) granulation of the compacted pieces; and (4) compaction of the granulated particles (Clere Declaration ¶ 6).

Example 2 in Shaffer I and II discloses a similar process as in Example 1 for preparing a compacted BN product, however, the crushed starting powder in Example 2 had an oxygen content of 0.60 %, a surface area of 3.02 m<sup>2</sup>/g, a tap density of 0.89 g/cc, 0.14 % soluble borates, and a particle size ranging from 44 µm (-325 mesh) to greater than 420 µm (+40 mesh) (see Shaffer I at col. 4, line 65 to col. 5, line 4 and Table II) (Clere Declaration ¶ 7). In addition, four additional granulation/compaction steps as described in Example 1 were performed (Shaffer I, col. 5, lines 4-5) (Clere Declaration ¶ 7). The resultant compact density was 1.91 g/cc (Shaffer I, col. 5, line 6) (Clere Declaration ¶ 7).

Example 3 in Shaffer I and II also discloses the same process as in Example 1 for preparing a compacted BN product, however, the crushed starting powder in Example 3 had an oxygen content of 0.275 %, a surface area of  $5.26 \text{ m}^2/\text{g}$ , a tap density of 0.85 g/cc, 0.10 % soluble borates, and a particle size ranging from  $44 \text{ }\mu\text{m}$  (-325 mesh) to greater than  $420 \text{ }\mu\text{m}$  (+40 mesh) (see Shaffer I at col. 5, lines 21-32 and Table II) (Clere Declaration ¶ 8). The resultant compact density was 1.89 g/cc (Shaffer I, col. 5, lines 31-32) (Clere Declaration ¶ 8).

Example 4 in Shaffer I and II discloses a similar process for preparing a compacted BN product, however, the crushed starting powder in Example 4 had an oxygen content of 0.60 %, a surface area of  $3.02 \text{ m}^2/\text{g}$ , a tap density of 0.89 g/cc, 0.14 % soluble borates, and a particle size ranging from  $44 \text{ }\mu\text{m}$  (-325 mesh) to greater than  $420 \text{ }\mu\text{m}$  (+40 mesh) (see Shaffer I at col. 5, line 37 to col. 6, line 13 and Table II) (this is the same starting powder as in Example 2) (Clere Declaration ¶ 9). The crushed powder was compacted at 19,000 psi using a uniaxial press, and was then formed into granules of 1/16 inch and finer using a granulator (Shaffer I, col. 6, lines 2-5) (Clere Declaration ¶ 9). The granules were once again compacted at 19,000 psi (Shaffer I, col. 6, lines 5-6) (Clere Declaration ¶ 9). The compacts were crushed using a sawtooth and roll crusher and screened through a 120 mesh screen, resulting in a powder having a tap density of  $0.68 \text{ m}^2/\text{g}$  (Shaffer I, col. 6, lines 6-8) (Clere Declaration ¶ 9).

In order to determine the thermal diffusivity of a product produced as described in Shaffer I and II, an experiment corresponding to Example 1 (i.e., including the four steps of Example 1) was conducted (Clere Declaration ¶ 10). In particular, BN was: (1) crushed and milled to -325 mesh with a roll mill to produce high purity hBN having an oxygen content of less than 1%, a surface area of 2 to  $4 \text{ m}^2/\text{g}$ , a tap density of approximately 0.28 g/cc, a  $\text{B}_2\text{O}_3$  content (soluble borate) of less than 0.1%, and a particle size ranging from  $44 \text{ }\mu\text{m}$  (-325 mesh) to greater than  $420 \text{ }\mu\text{m}$  (+40 mesh) (Clere Declaration ¶ 10). Although the tap density of the crushed BN in this experiment was lower than that described in Example 1 of Shaffer I and II, this modification would not result in a change in thermal diffusivity (Clere Declaration ¶ 10). In particular, thermal diffusivity is determined by the structure of the BN on the crystallite level (Clere Declaration ¶ 10). This structure is not affected by the tap density value (Clere Declaration ¶ 10). (2) The high purity hexagonal boron nitride was then isopressed at 19,000 psi into a compacted form (Clere Declaration ¶ 10). Although Shaffer I and II describe the use of a horizontal press or uniaxial press to produce the compacted form, compaction via isopressing would not result in a change in thermal diffusivity (Clere Declaration ¶ 10). The only difference would be that less alignment of particles would be achieved through the use of isopressing (Clere Declaration ¶ 10). The

thermal diffusivity of the compacted form was measured by laser flash technique (using an Appollo Laser, Model M22) and was found to be  $0.09 \text{ cm}^2/\text{sec}$  (Clere Declaration ¶ 10). (3) The compacted form was then crushed in a roll mill (Model 666 "F" Gran-U-Lizer, Modern Process Equipment, Chicago, IL) (Clere Declaration ¶ 10). Although Shaffer I and II describe granulating the compacted form, the substitution of crushing in a roll mill will not impact the resulting thermal diffusivity, as the structure of the BN on the crystallite level will not be changed by either granulating or crushing (Clere Declaration ¶ 10). (4) Finally, the crushed particles were isopressed again at 19,000 psi into a compacted form. The thermal diffusivity was measured by laser flash technique (using an Appollo Laser, Model M22) and was found to be  $0.10 \text{ cm}^2/\text{sec}$  (Clere Declaration ¶ 10).

Example 2 of Shaffer I and II relates to the use of additional cycles of granulation and compaction (Clere Declaration ¶ 11). Such additional cycles of crushing and compaction will not measurably change the thermal diffusivity of the resulting compact, since crushing and compaction will not change the crystallite structure of the BN (Clere Declaration ¶ 11). Moreover, the modifications in oxygen content, surface area, tap density, percent of soluble borates, and percentages of particle sizes in within the range of  $44 \text{ }\mu\text{m}$  (-325 mesh) to greater than  $420 \text{ }\mu\text{m}$  (+40 mesh) for the starting crushed BN used in Example 2, as compared to Example 1, would not measurably change the resulting thermal diffusivity value (Clere Declaration ¶ 11). In particular, these properties do not relate to the crystallite structure of the BN and, therefore, would not change the thermal diffusivity value obtained (Clere Declaration ¶ 11).

With regard to Example 3 of Shaffer I and II, an identical process to that described in Example 1 was used with a starting high purity BN having modifications in oxygen content, surface area, tap density, percent of soluble borates, and percentages of particle sizes in within the range of  $44 \text{ }\mu\text{m}$  (-325 mesh) to greater than  $420 \text{ }\mu\text{m}$  (+40 mesh) (Clere Declaration ¶ 12). As described above, such modifications in these properties would not measurably change the resulting thermal diffusivity value (Clere Declaration ¶ 12).

Finally, with regard to Example 4 of Shaffer I and II, a similar process to that described in Example 1 was used, however, a uniaxial press was used (as opposed to a horizontal press), granules of 1/16 inch and finer were produced using a granulator (as opposed to the use of a screen with 1/2 inch openings), and an additional crushing step was performed after the second compaction (Clere Declaration ¶ 13). The use of a different type of press, the production of finer granules, and an additional crushing step would not impact the resulting thermal diffusivity, as these processes would not impact the structure of the boron nitride on the crystallite level (Clere Declaration ¶ 13). Moreover, the starting high

purity BN used in Example 4 is identical to that used in Example 2, and as described in paragraph 11, would not measurably change the resulting thermal diffusivity value (Clere Declaration ¶ 13).

Accordingly, using the methods described in Shaffer I and II, a thermal diffusivity of  $0.10 \text{ cm}^2/\text{s}$  was obtained (see Clere Declaration ¶ 10) (Clere Declaration ¶ 14). This is below the value claimed in the present application (Clere Declaration ¶ 14). In contrast to the disclosure of Shaffer I and II, when applying the very dissimilar methodology described in the present application (i.e., sintering after the first isopressing) to the same starting powder described in Clere Declaration ¶ 10, a product having a measured thermal diffusivity of  $0.18 \text{ cm}^2/\text{s}$  was produced (Clere Declaration ¶ 14).

### *Corrigan*

Corrigan relates to a method for converting hBN particulates into cubic BN (cBN) particles or agglomerates (see Abstract) (Clere Declaration ¶ 15). This reference discloses that the starting hBN powder can be compressed into a billet form and granulated to give particles of a size larger than those commercially available (col. 3, lines 4-6) (Clere Declaration ¶ 15). In addition, Corrigan discloses that the hBN source powder may be densified or pre-pressed into an agglomerated mass or billet at low temperatures, and the mass or billet is then granulated into agglomerated particles of a desired size (col. 2, lines 48-51) (Clere Declaration ¶ 15). Corrigan also discloses that the hBN particles may be vacuum heated and fired, prior to compression and granulation, to remove volatile impurities (col. 3, lines 18-22) (Clere Declaration ¶ 15). In the Examples, the hBN powder is described as “fine-sized HBN powder . . . vacuum fired in the boron nitride thermal decomposition range . . . to obtain a source powder (col. 6, lines 11-14) (Clere Declaration ¶ 15).

No further information regarding the starting hBN material in Corrigan is provided (Clere Declaration ¶ 16). Accordingly, it is not possible to duplicate the disclosure in Corrigan to determine a thermal diffusivity value of the hBN material disclosed (Clere Declaration ¶ 16). However, as shown above with regard to the disclosure in Shaffer I and II, boron nitride powders will not necessarily have a thermal diffusivity of from about  $0.15 \text{ cm}^2/\text{s}$  to about  $0.20 \text{ cm}^2/\text{s}$  as claimed in the present application and sintering of a compacted body is required to obtain such a thermal diffusivity value (Clere Declaration ¶ 16). Sintering of a compacted body of hBN powder is not disclosed in Corrigan (Clere Declaration ¶ 16).

*Kawasaki*

Kawasaki relates to melamine borate particles, hBN particles and an hBN powder produced using the melamine borate particles, a resin or rubber composite containing the hBN powder, an hBN sintered body, and methods of making the above products (col. 1, lines 4-14) (Clere Declaration ¶ 17). This reference discloses formation of an hBN powder by mixing melamine borate particles, or a mixture containing them, and a crystallization catalyst and firing the mixture in a non-oxidizing gas atmosphere at from 1700 °C to 2200 °C (col. 7, lines 53-56 and col. 8, lines 28-32; Examples 1 and 3) (Clere Declaration ¶ 17). Kawasaki also discloses a method for producing hBN powder by calcining melamine borate particles, or a mixture containing them, to form amorphous BN or hBN, adding a crystallization catalyst, and firing the resulting mixture (col. 8, lines 32-39; Examples 2, 4, and 5) (Clere Declaration ¶ 17). This reference further distinguishes the hBN particles obtained in accordance with the invention of Kawasaki from conventional hBN particles, such as non-aggregated scaly particles, hBN blocks wherein crystals are undeveloped, granulated particles, or particles obtained by pulverizing an hBN sintered body (col. 5, line 27 to col. 6, line 3) (Clere Declaration ¶ 17).

For comparison to the Kawasaki patent, the thermal diffusivity of a melamine and boric acid derived BN powder was investigated (Clere Declaration ¶ 18). Melamine (46 kg) and boric acid (54 kg) were dry mixed and compacted into briquettes (Clere Declaration ¶ 18). These briquettes were heated in an inconel tube furnace to 950 °C under a nitrogen atmosphere to form amorphous BN (see Examples 2, 4, and 5 of Kawasaki) (Clere Declaration ¶ 18). This amorphous BN was then heated to 2000 °C in an induction furnace under a nitrogen atmosphere to form hBN (Clere Declaration ¶ 18). The thermal diffusivity was measured by laser flash technique (using an Appollo Laser, Model M22) and was found to be 0.39 cm<sup>2</sup>/s (Clere Declaration ¶ 18). This high thermal diffusivity can be attributed to the high degree of agglomeration and crystallinity formed during processing of the powder above 950 °C (Clere Declaration ¶ 18).

A similar thermal diffusivity value (i.e., greater than 0.20 cm<sup>2</sup>/s) will be obtained using the analogous method described in Kawasaki (Clere Declaration ¶ 19). In particular, the material produced by the teachings of Kawasaki, in terms of thermal diffusivity, will not differ from the material produced using melamine and boric acid as described in Clere Declaration ¶ 18, because the crystallinity of the BN produced in both cases will be similar (Clere Declaration ¶ 19). More specifically, in Kawasaki melamine diborate is formed and fired at temperatures of 1700-2100 °C (Examples 1-5, col. 11, line 56 to col. 13, line 34 and Tables 2-6) (Clere Declaration ¶ 19). In Examples 2, 4, and 5 of

Kawasaki, the melamine diborate mixture is calcined at 800 °C or 1600 °C to form amorphous boron nitride prior to heating to form hBN (Clere Declaration ¶ 19). Thus, a different feed material is used in Kawasaki than in the experiment we conducted as described in Clere Declaration ¶ 18 (Clere Declaration ¶ 19). However, the crystal structure of hBN formed at high temperature (above 1600 °C) is independent of the feed materials (Clere Declaration ¶ 19). This is illustrated by a comparison of x-ray diffraction patterns for the material produced as described in Clere Declaration ¶ 18 (Exhibits A and B to the Clere Declaration) and the x-ray diffraction patterns for a material produced using melamine diborate as a starting material (Exhibit C to the Clere Declaration) (Clere Declaration ¶ 19). In particular, Exhibit A shows the x-ray diffraction pattern for the starting material used in the experiment described in Clere Declaration ¶ 18 (Clere Declaration ¶ 19). This corresponds to Figure 1(a) of Sato et al., “Microstructural Development with Crystallization of Hexagonal Boron Nitride,” J. Material Sci. Letters, 16(10):795-798 (1997) (“Sato”) attached as Exhibit C to the Clere Declaration, which relates to a starting BN material produced from melamine diborate (Clere Declaration ¶ 19). The two x-ray diffraction patterns (Exhibit A and Figure 1(a)) show the same two broad peaks (Clere Declaration ¶ 19). Exhibit B shows the x-ray diffraction pattern for the resulting hBN produced in the experiment described in Clere Declaration ¶ 18 (Clere Declaration ¶ 19). This corresponds to Figure 1(e) of Sato attached as Exhibit C to the Clere Declaration, which relates to a resulting hBN product using melamine diborate as a starting material and heating at 2000 °C (Clere Declaration ¶ 19). The two x-ray diffraction patterns (Exhibit B and Figure 1(e)) again show the presence of the same peaks (Clere Declaration ¶ 19). Thus, the crystal structure of the two products is the same, although the starting materials and reaction conditions are different (Clere Declaration ¶ 19). More specifically, the materials are identical, chemically and structurally at the atomic level, due to their treatment at high temperature (above 1600 °C) (Clere Declaration ¶ 19). The same is true of the hBN produced by the method described in Kawasaki as compared to the hBN produced as described in Clere Declaration ¶ 18 -- both products are produced by treatment at high temperature (above 1600 °C) (Clere Declaration ¶ 19). Therefore, properties of the hBN produced in Kawasaki and the hBN produced in the experiment described in Clere Declaration ¶ 18, such as crystallinity, thermal diffusivity, and electrical conductivity, will be the same (Clere Declaration ¶ 19). Moreover, the addition of the crystallization catalysts disclosed in Kawasaki merely impacts the rate of reaction and would not alter the crystal structure, and, therefore, the thermal diffusivity, of the resulting product (Clere Declaration ¶ 19).

Accordingly, hBN with a thermal diffusivity value greater than 0.20 cm<sup>2</sup>/s will be produced according to the methods of Kawasaki (Clere Declaration ¶ 20).

As a result, none of Shaffer I, Shaffer II, Corrigan, or Kawasaki disclose or suggest an hBN powder having a thermal diffusivity between  $0.15 \text{ cm}^2/\text{s}$  to about  $0.20 \text{ cm}^2/\text{s}$  or the methodology for producing an hBN powder having a thermal diffusivity between  $0.15 \text{ cm}^2/\text{s}$  to about  $0.20 \text{ cm}^2/\text{s}$ , as required by the claims of the present application. Therefore, this rejection is improper and should be withdrawn.

The rejection of claims 13-16 under 35 U.S.C. § 102(b) as being anticipated by a public use or sale as described in Quotation (June 5, 1998) and Request for Quotation Form (May 15, 1998) ("1998 Quotation") and Quotation (December 2, 1999) and Request for Quotation Form for Item 1, Item 2, and Item 3 (November 30, 1999) ("1999 Quotation") is respectfully traversed.

In particular, the 1998 Quotation is a redacted quotation and request for quotation form for a boron nitride powder having an estimated thermal diffusivity of  $0.1819 \text{ cm}^2/\text{s}$  and an average particle size of about 150 microns to about 300 microns, resulting from a May 15, 1998, phone call requesting a quotation. An experimental sample had been first provided to the party identified in the 1998 Quotation, at no charge, on May 8, 1998, pursuant to a confidentiality agreement. However, the 1998 Quotation neither discloses nor suggests "[a] boron nitride powder having a thermal diffusivity of from about  $0.15 \text{ cm}^2/\text{s}$  to about  $0.20 \text{ cm}^2/\text{s}$  and an average particle size of from about  $2 \text{ }\mu\text{m}$  to about  $20 \text{ }\mu\text{m}$ ," as required by claim 13 (and its dependent claims 14-16) (emphasis added).

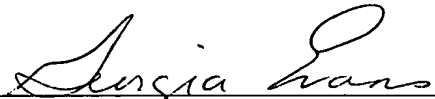
The 1999 Quotation is a redacted quotation and request for quotation form for Item 1, a boron nitride powder having an estimated thermal diffusivity of  $0.1819 \text{ cm}^2/\text{s}$  and an average particle size of about 150 microns to about 300 microns, Item 2, a boron nitride powder having an estimated thermal diffusivity of  $0.1819 \text{ cm}^2/\text{s}$  and an average particle size of from about 150 microns to about 300 microns, and Item 3, a boron nitride powder having an estimated thermal diffusivity of above  $0.25 \text{ cm}^2/\text{s}$  and an average particle size of from about 60 microns to about 125 microns. However, the 1999 Quotation neither discloses nor suggests "[a] boron nitride powder having a thermal diffusivity of from about  $0.15 \text{ cm}^2/\text{s}$  to about  $0.20 \text{ cm}^2/\text{s}$  and an average particle size of from about  $2 \text{ }\mu\text{m}$  to about  $20 \text{ }\mu\text{m}$ ," as required by claim 13 (and its dependent claims 14-16) (emphasis added).

Accordingly, the rejection based on the 1998 Quotation and the 1999 Quotation is improper and should be withdrawn.

In view of all of the foregoing, applicant submits that this case is in condition for allowance and such allowance is earnestly solicited.

Respectfully submitted,

Dated: December 19, 2003

  
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